

Los Alamos National Laboratory
Environmental Restoration Program
Standard Operating Procedure

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Total Organic Carbon

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TOTAL ORGANIC CARBON

1.0 PURPOSE

This procedure describes the method of determining the ash content and the total organic carbon of soil samples by air drying, heating within an oven or furnace, or a combination thereof.

2.0 SCOPE

2.1 Applicability

This procedure is applicable to all personnel determining total organic content for the Environmental Restoration program.

2.2 Training

Field team members using this procedure must document that they have read and understand this procedure and the procedures in Section 1.0, General Instructions.

3.0 DEFINITIONS

There are no unique definitions in this procedure.

4.0 BACKGROUND AND/OR CAUTIONS

Refer to the attached ASTM standard method D2974-87 and to the Site Health and Safety plan for hazards and/or cautions.

5.0 EQUIPMENT

Refer to the attached ASTM standard method D2974-87.

6.0 PROCEDURE

Refer to the attached ASTM standard method D2974-87.

7.0 REFERENCES

The following procedures are directly associated with this procedure and should be reviewed prior to field operation:

ASTM STANDARD D2974-87. 1987. Standard Test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils.

LANL-ER-SOPs in Section 1.0, General Instructions.

8.0 RECORDS

The records generated by this procedure will include the Chain-of-Custody/Request for Analysis form (SOP-01.04) which will include sample collection information logs, and calculations. Also included will be the laboratory report with written calculations, furnace temperature, and whether moisture contents are by proportion of as-received mass (wet mass) or by oven-dried mass.

9.0 ATTACHMENTS

- A. ASTM STANDARD D2974-87. 1987. Standard Test Method for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils.

ASTM STANDARD D2974-87, STANDARD TEST METHOD FOR MOISTURE, ASH, AND ORGANIC MATTER OF PEAT AND OTHER ORGANIC SOILS



Designation: D 2974 - 87

Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils¹

This standard is issued under the fixed designation D 2974; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the measurement of moisture content, ash content, and organic matter in peats and other organic soils, such as organic clays, silts, and mucks.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Methods

2.1 *Method A*—Moisture is determined by drying a peat or organic soil sample at 105°C. The moisture content is expressed either as a percent of the oven dry mass or of the as-received mass.

2.2 *Method B*—This is an alternative moisture method which removes the total moisture in two steps: (1) evaporation of moisture in air at room temperature (air-drying), and (2) the subsequent oven drying of the air-dried sample at 105°C. This method provides a more stable sample, the air-dried sample, when tests for nitrogen, pH, cation exchange, and the like are to be made.

2.3 *Methods C and D*—Ash content of a peat or organic soil sample is determined by igniting the oven-dried sample from the moisture content determination in a muffle furnace at 440°C (Method C) or 750°C (Method D). The substance remaining after ignition is the ash. The ash content is expressed as a percentage of the mass of the oven-dried sample.

2.4 Organic matter is determined by subtracting percent ash content from one hundred.

3. Apparatus

3.1 *Oven*, capable of being regulated to a constant temperature of 105 ± 5°C.

NOTE—The temperature of 105°C is quite critical for organic soils. The oven should be checked for "hot spots" to avoid possible ignition of the specimen.

3.2 *Muffle Furnace*, capable of producing constant temperatures of 440°C and 750°C.

¹ These test methods are under the jurisdiction of ASTM Committee D-18 on Soil and Rock and are the direct responsibility of Subcommittee D18.18 on Peats and Related Materials.

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3.3 *Evaporating Dishes*, of high silica or porcelain of not less than 100-mL capacity.

3.4 *Blender*, high-speed.

3.5 *Aluminum Foil*, heavy-duty.

3.6 *Porcelain Pan, Spoons*, and equipment of the like.

3.7 *Desiccator*.

4. Preparation of Sample

4.1 Place a representative field sample on a square rubber sheet, oil cloth, or equivalent material. Reduce the sample to the quantity required by quartering and place in a moisture-proof container. Work rapidly to prevent moisture loss or perform the operation in a room with a high humidity.

MOISTURE CONTENT

5. Method A

5.1 Record to the nearest 0.01 g the mass of a high silica or porcelain evaporating dish fitted with a heavy-duty aluminum foil cover. The dish shall have a capacity of not less than 100 mL.

5.2 Mix thoroughly the representative sample and place a test specimen of at least 50 g in the container described in 5.1. Crush soft lumps with a spoon or spatula. The thickness of peat in the container should not exceed 3 cm.

5.3 Cover immediately with the aluminum foil cover and record the mass to the nearest 0.01 g.

5.4 Dry uncovered for at least 16 h at 105°C or until there is no change in mass of the sample after further drying periods in excess of 1 h. Remove from the oven, cover tightly, cool in a desiccator, and record the mass.

6. Method A Calculation

6.1 Calculate the moisture content as follows:

$$\text{Moisture Content, \%} = [(A - B) \times 100]/A$$

where:

A = mass of the as-received test specimen, g, and

B = mass of the oven-dried specimen, g.

6.1.1 This calculation is used primarily for agriculture, forestry, energy, and horticultural purposes and the result should be referred to as the moisture content as a percentage of as-received or total mass.

6.2 An alternative calculation is as follows:

$$\text{Moisture Content, \%} = [(A - B) \times 100]/B$$

where:

A = as-received test specimen, g, and

B = mass of the oven-dried specimen, g.

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6.2.1 This calculation is used primarily for geotechnical purposes and the result should be referred to as the moisture content as a percentage of oven-dried mass.

6.3 Take care to indicate the calculation method used.

7. Method B

7.1 This method should be used if pH, nitrogen content, cation exchange capacity, and the like are to be tested.

7.2 Mix the sample thoroughly and select a 100 to 300 g representative sample. Determine the mass of this sample and spread evenly on a large flat pan. Crush soft lumps with a spoon or spatula and let the sample come to moisture equilibrium with room air. This will require at least 24 h. Stir occasionally to maintain maximum air exposure of the entire sample. When the mass of the sample reaches a constant value, calculate the moisture removed during air drying as a percentage of the as-received mass.

7.3 Grind a representative portion of the air-dried sample for 1 to 2 min in a high-speed blender. Use the ground portion for moisture, ash, nitrogen, cation exchange capacity tests, and the like.

7.4 Thoroughly mix the air-dried, ground sample. Weigh to the nearest 0.01 g the equivalent of 50 g of test specimen on an as-received basis. Determine the amount, in grams, of air-dried sample equivalent to 50 g of as-received sample, as follows:

$$\text{Equivalent Sample Mass, g} = 50.0 - [(50 \times M)/100]$$

where:

M = moisture removed in air drying, %.

7.5 Place the sample in a container as described in 5.1 and proceed as in Method A.

8. Method B Calculation

8.1 Calculate the moisture content as follows:

$$\text{Moisture Content, \%} = (50 - B) \times 2$$

where:

B = oven-dried sample, g.

8.1.1 This calculation gives moisture content as a percentage of as-received mass.

8.2 An alternative calculation is as follows:

$$\text{Moisture Content, \%} = [(50 - B) \times 100]/B$$

8.2.1 This calculation gives moisture content as a percentage of oven-dried mass.

ASH CONTENT

9. Method C

9.1 Determine the mass of a covered high-silica or porcelain dish.

9.2 Place a part of or all of the oven-dried test specimen from a moisture determination in the dish and determine the mass of the dish and specimen.

9.3 Remove the cover and place the dish in a muffle furnace. Gradually bring the temperature in the furnace to 440°C and hold until the specimen is completely ashed (no change of mass occurs after a further period of heating).

9.4 Cover with the retained aluminum foil cover, cool in a desiccator, and determine the mass.

9.5 This method should be used for all geotechnical and general classification purposes.

10. Method D

10.1 Determine the mass of a covered high-silica or porcelain dish.

10.2 Place a part of or all of the oven-dried test specimen from a moisture determination in the dish and determine the mass of the dish and specimen.

10.3 Remove the cover and place the dish in a muffle furnace. Gradually bring the temperature in the furnace to 750°C and hold until the specimen is completely ashed (no change of mass occurs after a further period of heating).

10.4 Cover with the retained aluminum foil cover, cool in a desiccator, and determine the mass.

10.5 This method should be used when peats are being evaluated for use as a fuel.

11. Calculation for Methods C and D

11.1 Calculate the ash content as follows:

$$\text{Ash Content, \%} = (C \times 100)/B$$

where:

C = ash, g, and

B = oven-dried test specimen, g.

ORGANIC MATTER

12. Calculation

12.1 Determine the amount of organic matter by difference, as follows:

$$\text{Organic matter, \%} = 100.0 - D$$

where:

D = ash content, %.

13. Report

13.1 Report the following information:

13.1.1 Results for organic matter and ash content, to the nearest 0.1 %.

13.1.2 Furnace temperature used for ash content determinations.

13.1.3 Whether moisture contents are by proportion of as-received mass or oven-dried mass.

13.1.3.1 Express results for moisture content as a percentage of as-received mass to the nearest 0.1 %.

13.1.3.2 Express results for moisture content as a percentage of oven-dried mass as follows:

(a) Below 100 % to the nearest 1 %.

(b) Between 100 % and 500 % to the nearest 5 %.

(c) Between 500 % and 1000 % to the nearest 10 %.

(d) Above 1000 % to the nearest 20 %.

14. Precision and Bias

14.1 The precision and bias of these test methods have not been determined. Data are being sought for use in developing a precision and bias statement.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.